

**PERCHLORATE in WATER by ION CHROMATOGRAPHY**  
Modified <sup>a</sup> EPA Method 300.0 (Revision 2.1, August 1993)

**Table 1.      Summary of Contract Required Detection Limits, Holding Times, and Preservation for Perchlorate**

Analytical Parameter	Contract Required Detection Limit (CRDL)	Technical and Contract Holding Times	Preservation
Perchlorate	5.0 µg/L	Technical: 28 days from collection; Contract: 21 days from receipt at laboratory	Cool to 4°C ±2°C

<sup>a</sup> EPA Method 300.0 modified for the analysis of perchlorate in water as described in the California Department of Health Services (DHS) method (Sanitation & Radiation Laboratories Branch; Determination of Perchlorate by Ion Chromatography, Rev. No. 0, June 3, 1997).

**Data Calculations and Reporting Units:**

Calculate the sample results according to Section 12 of EPA Method 300.0 (Revision 2.1, August 1993) or Section 12 of the California DHS method. Report sample results in the concentration unit of micrograms per liter (µg/L). Report perchlorate concentrations which are  $\geq 10$  µg/L to three significant figures and perchlorate concentrations which are  $< 10$  µg/L to two significant figures.

For rounding results, adhere to the following rules:

- a) If the number following those to be retained is less than 5, round down;
- b) If the number following those to be retained is greater than 5, round up; or
- c) If the number following the last digit to be retained is equal to 5, round down if the digit is even, or round up if the digit is odd.

All records of analysis and calculations must be legible and sufficient to recalculate all sample concentrations and QC results. Include an example calculations in the data package.

**Table 2. Summary of Calibration Procedures for Perchlorate by Modified EPA Method 300.0**

Calibration Element	Frequency	Acceptance Criteria	Corrective Action
Initial Calibration (minimum blank + 5 points for perchlorate)(ICAL) <sup>a, b</sup>	Initially; monthly; or whenever required due to failure of IPC	$r \geq 0.995$	1. Terminate analysis 2. Recalibrate and reanalyze sample
Instrument Performance Check (IPC) <sup>c</sup> (Separate source from ICAL standards)	Following the calibration and prior to sample analysis; after every 10 samples; and end of run	$\pm 10\%$ from expected concentration	1. Recalibrate and reanalyze sample 2. Reanalyze sample if good IPC
Retention time evaluation for IPC standard	Each analysis of IPC standard	$\pm 10\%$ from expected retention time	1. Recalibrate and reanalyze sample 2. Reanalyze sample if good IPC
Calibration Blank Verification (ICB, CCB)	After ICAL; every IPC; and end of the analytical sequence	$< \text{CRDL}$	1. Terminate analysis 2. Identify and correct problem 3. Recalibrate, verify, and reanalyze sample 4. All associated standards
CRDL Verification Standard	After initial IPC/CCB	$\pm 20\%$ from expected concentration	1. Reprep and reanalyze sample 2. Recalibrate and reanalyze sample

<sup>a</sup> The low level standard should be at a concentration equal to the contract required detection limit (CRDL).

<sup>b</sup> Report the retention time window for each analyte. Determine retention time windows as  $\pm 10\%$  of the mean retention time for each analyte in the calibration standards.

<sup>c</sup> The IPC standard solution should contain perchlorate at a concentration different from the concentration of perchlorate in the calibration standards.

**Table 3. Summary of Internal Quality Control Procedures for Perchlorate by Modified EPA Method 300.0**

QC Element	Frequency	Acceptance Criteria	Corrective Action
Laboratory Reagent Blank (LRB)	One per Batch or SDG <sup>a</sup> (1 per 20 samples minimum)	< CRDL	1. If lowest sample c than 10X the blank co 2. If samples are non 3. If detected sample less than 10X blank c samples must be prepa another method blank
Duplicate Sample (DUP)	One per batch or SDG (1 per 20 samples minimum)	RPD <20% for samples >5X CRDL; ± CRDL for samples <5X CRDL	1. Flag associated da
Laboratory Fortified Matrix (LFM) <sup>b</sup>	One per batch or SDG (1 per 20 samples minimum)	± 25% from expected value	1. Flag associated da
Laboratory Fortified Blank (LFB)	One per batch or SDG (1 per 20 samples minimum)	± 10% from expected concentration	1. Terminate analysis 2. Identify and docum 3. Reanalyze all asso

<sup>a</sup> SDG - Sample Delivery Group - each case of field samples received; or each 20 field samples within a case; or each 14 calendar day period during which field samples in a case are received.

<sup>b</sup> If the LFM sample exceeds the calibration range, the sample must be diluted appropriately, re-spiked, and reanalyzed.

Dilute and reanalyze samples with concentrations exceeding the range of the calibration curve. Results for such reanalyses should fall within the mid-range of the calibration curve. Report results and submit documentation for both analyses.

Perform confirmatory techniques, such as sample dilution and spiking, when the identification of a peak in the chromatogram is questionable. Spike the sample with an appropriate amount of the relevant standard and reanalyze.

Analyze a laboratory blank after the analysis of an unusually concentrated sample to check for contamination by carry-over. Any sample with perchlorate present at a concentration 2× the calibration range is considered an unusually concentrated sample.